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Abstract: PURPOSE In testing adhesion using shear bond test, a combination of shear and tensile forces occur at the interface, resulting in complex stresses. The jig designs used for this kind of test show variations in published studies, complicating direct comparison between studies. This study evaluated the effect of different jig designs on metal-ceramic bond strength and assessed the stress distribution at the interface using finite element analysis (FEA). MATERIALS AND METHODS Metal-ceramic (Metal: Ni-Cr, Wiron 99, Bego; Ceramic: Vita Omega 900, Vita) specimens (N = 36) (diameter: 4 mm, veneer thickness: 4 mm; base diameter: 5 mm, thickness: 1 mm) were fabricated and randomly divided into three groups (n = 12 per group) to be tested using one of the following jig designs: (a) chisel (CH) (ISO 11405), (b) steel strip (SS), (c) piston (PI). Metal-ceramic interfaces were loaded under shear until debonding in a universal testing machine (0.5 mm/min). Failure types were evaluated using scanning electron microscopy (SEM). FEA was used to study the stress distribution using different jigs. Metal-ceramic bond strength data (MPa) were analyzed using ANOVA and Tukey's tests ($\alpha = 0.05$). RESULTS The jig type significantly affected the bond results ($p = 0.0001$). PI type of jig presented the highest results (MPa) ($p < 0.05$) (58.2 ± 14.8), followed by CH (38.7 ± 7.6) and SS jig type (23.3 ± 4.2) ($p < 0.05$). Failure types were exclusively a combination of cohesive failure in the opaque ceramic and adhesive interface failure. FEA analysis indicated that the SS jig presented slightly more stress formation than with the CH jig. The PI jig presented small stress concentration with more homogeneous force distribution compared to the CH jig where the stress concentrated in the area where the force was applied. CONCLUSION Metal-ceramic bond strength was affected by the jig design. Accordingly, the results of in vitro studies on metal-ceramic adhesion should be evaluated with caution. CLINICAL SIGNIFICANCE When adhesion of ceramic materials to metals is evaluated in in vitro studies, it should be noted that the loading jig type affects the results. Clinical observations should report on the location and type of ceramic fractures in metal-ceramic reconstructions so that the most relevant test method can be identified.

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Effect of Jig Design and Assessment of Stress Distribution in Testing Metal-Ceramic Adhesion

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Short title: *Effect of testing conditions on metal-ceramic adhesion*

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Abstract

Purpose: In testing metal-ceramic adhesion using shear bond test, a combination of shear and tensile forces occur at the interface resulting in complex stresses. The jig designs used for this kind of test show variations in the published studies that complicate the direct comparison between different studies. This study evaluated the effect of different jig designs on metal-ceramic bond strength and assessed the stress distribution at the interface using finite element analysis.

Materials and Methods: Metal-ceramic (Metal: Ni-Cr, Wiron 99, Bego; Ceramic: Vita Omega 900, Vita) specimens (N=36) (diameter: 4 mm, veneer thickness: 4 mm; base diameter: 5 mm and thickness: 1 mm) were fabricated and randomly divided into three groups (n=12 per group) to be tested using one of the following jig designs: **a)** chisel (CH) (ISO 11405), **b)** steel strip (SS), **c)** piston (PI). Metal-ceramic interfaces were loaded under shear until debonding in a Universal Testing Machine (0.5 mm/min). Failure types were evaluated using Scanning Electron Microscopy (SEM). Finite element analysis (FEA) was used to study the stress distribution using different jigs. Metal-ceramic bond strength data (MPa) were analyzed using ANOVA and Tukey's tests ($\alpha=0.05$).

Results: The jig type significantly affected the bond results ($p = 0.0001$). PI type of jig presented the highest results (MPa) ($p < 0.05$) (58.2 ± 14.8) followed by CH (38.7 ± 7.6) and SS jig type (23.3 ± 4.2) ($p < 0.05$). Failure types were exclusively a combination of cohesive failure in the opaque ceramic and adhesive interface failure. FEA analysis indicated that SS jig presented slightly more stress formation than with CH jig. PI jig presented small stress concentration with more homogeneous force distribution compared to CH jig where the stress concentrated in the area where the force was applied.

Conclusion: Metal-ceramic bond strength was affected by the jig design. Accordingly, the results of in vitro studies on metal-ceramic adhesion should be evaluated with caution.

Clinical Significance: When adhesion of ceramic materials to metals is evaluated in in vitro studies, it should be noted that the loading jig type affects the results. Clinical observations should report on the

location and type of ceramic fractures in metal-ceramic reconstructions so that the most relevant test method could be identified.

Keywords Adhesion; Bond testing; Dental porcelain; Finite element analysis; Shear bond test.

Introduction

Clinically, one of the main problems in metal-ceramic fixed dental prosthesis (FDP) is related to the adhesion between the ceramic veneer and the metallic frameworks leading to fracture of the ceramics with or without metal exposure.¹⁻⁴ The quality of the metal-ceramic restoration union is frequently verified by different laboratory tests such as flexural, tensile and shear tests.^{3,5-9} The results of such studies may not be directly extrapolated to clinical situations but they could be considered as useful tools in establishing a clinical study protocol, previewing the effectiveness and behaviour of metal-ceramic combinations to be used. Moreover, the constant modifications in the material composition are another complication factor. Prior to a clinical study, in vitro studies help ranking performance of material combinations. Therefore, laboratory methods that take less time to be executed and permit evaluating the bond strength of metal-ceramic interfaces are important. In that respect, shear bond tests are the most popular in dental literature.^{5-8,10-12} Forces are applied on a structure creates stresses that express how these forces are distributed in a given a structure. In that regard, forces that are applied laterally on the specimens create the complex stresses at the interface. Published studies use various jig designs that cause variations in the resulting values that complicate the direct comparison between different studies.

The objectives of this study therefore were to evaluate the effect of different jig designs on metal-ceramic bond strength and assess the stress distribution at the interface using finite element analysis. The null hypothesis tested was that jig type used for testing metal-ceramic shear bond would not affect the results significantly.

Materials and Methods

Standard (diameter: 4 mm, veneer thickness: 4 mm; base diameter: 5 mm and thickness: 1 mm) wax patterns (Occlusal Wax Bego, Bremen, Germany) were prepared using a metallic matrix and cast in an induction machine (Ducatron, 3 series, Ugin Dentaire, France) using Ni-Cr alloy (Wiron 99, Bego). After casting, metal specimens were air-abraded with 110 μm Al_2O_3 (Korox, Bego) at 3 bar from a distance of 20

mm at 45° for 10 seconds. They were then ultrasonically cleaned in distilled water (Vitasonic II, Vita, Bad Säckingen, Germany) for 10 minutes and dried. The ceramic was applied on the air-abraded surfaces of the cast specimens.

Two layers of opaque ceramic (Vita Omega 900 Opaque) were applied in a controlled manner as described elsewhere¹³ and fired in a ceramic oven (Vacumat 40, Vita). Subsequently, dentin ceramic (Vita Omega 900 Dentin, Vita) was applied and fired in two steps to minimize the shrinkage according to manufacturer's instructions. No glaze ceramic was applied on the specimen surfaces. After firing the ceramic, the quality of the interface and the external form of the specimens were visually ensured that there is a smooth junction. If defects were noted, new specimens were fabricated. All specimens were then seated in the jig to verify their adaptation.

The specimens (N=36) (diameter: 4 mm, veneer thickness: 4 mm) were randomly divided into three groups (n=12 per group) to be tested using one of the following jig design: **a)** chisel (CH) (ISO 11405),¹⁴ **b)** steel strip (SS), **c)** piston (PI) (Figs. 1a-c).

Metal-ceramic interfaces were loaded under shear until debonding in a Universal Testing Machine (EMIC, Model DL 1000, São José dos Pinhais, Brazil) with a load cell of 500 kg at a cross-head speed of 0.5 mm/min.

Failure types were evaluated using Scanning Electron Microscopy (SEM) (JSM-5500, Jeol, Tokyo, Japan).

Finite element analysis (FEA) was used to study the von Mises stress distribution under different loading conditions. For FEA, material properties were considered linear. A geometric model was built (IDEAS, UGSPLM Solutions, Plano, TX, USA) considering the specimen dimensions.

For material properties, the following were considered for FEA analysis: elasticity modulus of Ni-Cr: 205 GPa, Poisson's coefficient: 0.33, elasticity modulus of ceramic: 68.9 GPa, Poisson's coefficient: 0.28. The

manufacturers provided these data. The loading was performed, simulating the conditions to which the specimens were submitted, using the three jigs. The 3-D FEA were constructed with 1884 nodes and 1570 elements, and a load force of 600 N was used to make the analysis. The loading was performed replicating the laboratory conditions of this study. The blade was set to a point, contacting the ceramic and propagating at the bonding interface. While for the SS, it was considered that the strip surrounded the ceramic 180 degrees, for PI, load was applied to the ceramic including its margins.

Metal-ceramic bond strength data (MPa) were analyzed using ANOVA and Tukey's tests ($\alpha=0.05$).

Results

Metal-ceramic bond strength was significantly affected when tested with different jig types ($p = 0.0001$). PI type of jig presented the highest results (MPa) ($p < 0.05$) followed by CH and SS jig type ($p < 0.05$) (Fig. 2).

Failure types were exclusively a combination of cohesive failure in the opaque ceramic and adhesive interface failure. In the CH (Figs. 3a-c) failure occurred initially in the ceramic, at the contact area with the chisel but ceramic remnants were less in the PI. When SS jig was used, larger quantity of ceramic remained on the metal in the form of a half-moon fracture (Figs. 3d-f). With the PI, failure consistently initiated from the opaque ceramic-metal interface with extensive areas of metal indicating adhesive failure at the opaque ceramic-metal interface (Figs. 3h-i).

FEA analysis indicated greater stress concentration in a small area when shear bond strength test was performed by CH jig compared to other groups, followed by SS and PI (Figs. 4a-c). PI jig presented small stress concentration with more homogeneous force distribution at both compressive and tensile zones (Fig. 4c) compared to CH jig where the stress concentrated in the area where the force was applied only (Fig. 4a).

Discussion

Since different jig designs used to test metal-ceramic bond strength showed significant differences, the null hypothesis could be rejected. There is inconsistency in measuring the bond strength of the veneering ceramics to coping substrates and an effort for its standardization is needed. While Lombardo et al.⁹ recommends a cross-head speed between 0.45 and 1.05 mm/min for the shear bond strength test, Hara et al.¹⁵ recommended 0.5 to 0.75 mm/min. Tests performed at elevated cross-head speed could generate unfavourable distribution of the stresses, resulting in cohesive failures in the ceramic that may not allow measuring the real adhesion at the interface. In this study, in order to be able to make comparisons with previous studies, cross-head speed above 1 mm/min was not practiced at the interface.¹⁵ In fact, during mastication displacement rates can easily exceed this value.¹⁵ Thus, future studies should correlate the findings metal-ceramic adhesion tests with the clinical observations especially in failure cases. Nevertheless, the mean bond strength results of the present study from the PI group (58.2 ± 14.8) were slightly less (61.3 ± 8.4 MPa) than that of Vasquez et al.¹³ where the same jig type and ceramic were used but in combination with Au-Pd alloy. The difference could be attributed to lower elastic modulus of AuPd versus NiCr where the latter possibly allowed for less deflexion and thereby, debonding of the ceramic at a lower magnitude of force.

The variation coefficients found in the current study were of approximately 18 to 25% (CH: 19.69; SS: 18.06; PI: 25.51), which is pursuant to the ISO standard that indicates a coefficient with a variation less than 50%. In general, the variation coefficients of the shear bond tests vary from 20 to 60%.¹

FEA analysis indicated higher stress concentration with the CH jig followed by SS where the latter should be considered a better test in terms of stress distribution when compared to CH. Among all jig types, SS and PI presented a more homogeneous stress distribution. Most probably the loading jig produced no fulcrum on the ceramic cylinder and thereby, no superficial deflection. In this case, the tensile and compression forces produced perpendicularly at the interface were smaller than the ones obtained in CH systems.

With the CH jig, the knife blade may create shear and bending and thus the stress distribution at the interface is not the presumed shear bond strength. At this point, the ceramic was fractured and the cleavage propagation reached the interface, forming tension and compression, finally causing the deflection. In addition, it is almost impossible to achieve homogenous forces at the interface that is in fact also the clinical situation in the majority of the cases.

The lowest results were obtained with the SS jig indicating that the highest stresses occurred at the interface increases as the distance between the loading application point and the bond surface also increases; this is explained by the deformation of the stainless steel strip after tests.¹⁶⁻¹⁸

Considering that the failure types commonly observed clinically are between metal and the opaque layer, it can be stated that all jig types tested, despite the variations in the measured bond strength values, demonstrated clinically relevant failure types.⁴

Conclusions

1. When adhesion of veneering ceramic to NiCr is tested, shear bond strength tested by piston type of jig presented the highest shear bond strength followed by chisel.
2. Finite element analysis demonstrated that piston jig and steel strip presented less stress concentration with more homogeneous force distribution compared to chisel type.
3. Failure types were exclusively a combination of cohesive failure in the opaque ceramic and adhesive failure between the metal and opaque ceramic indicating the weakest link between the metal and the opaque ceramic.

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Captions to tables and legends:

Figures:

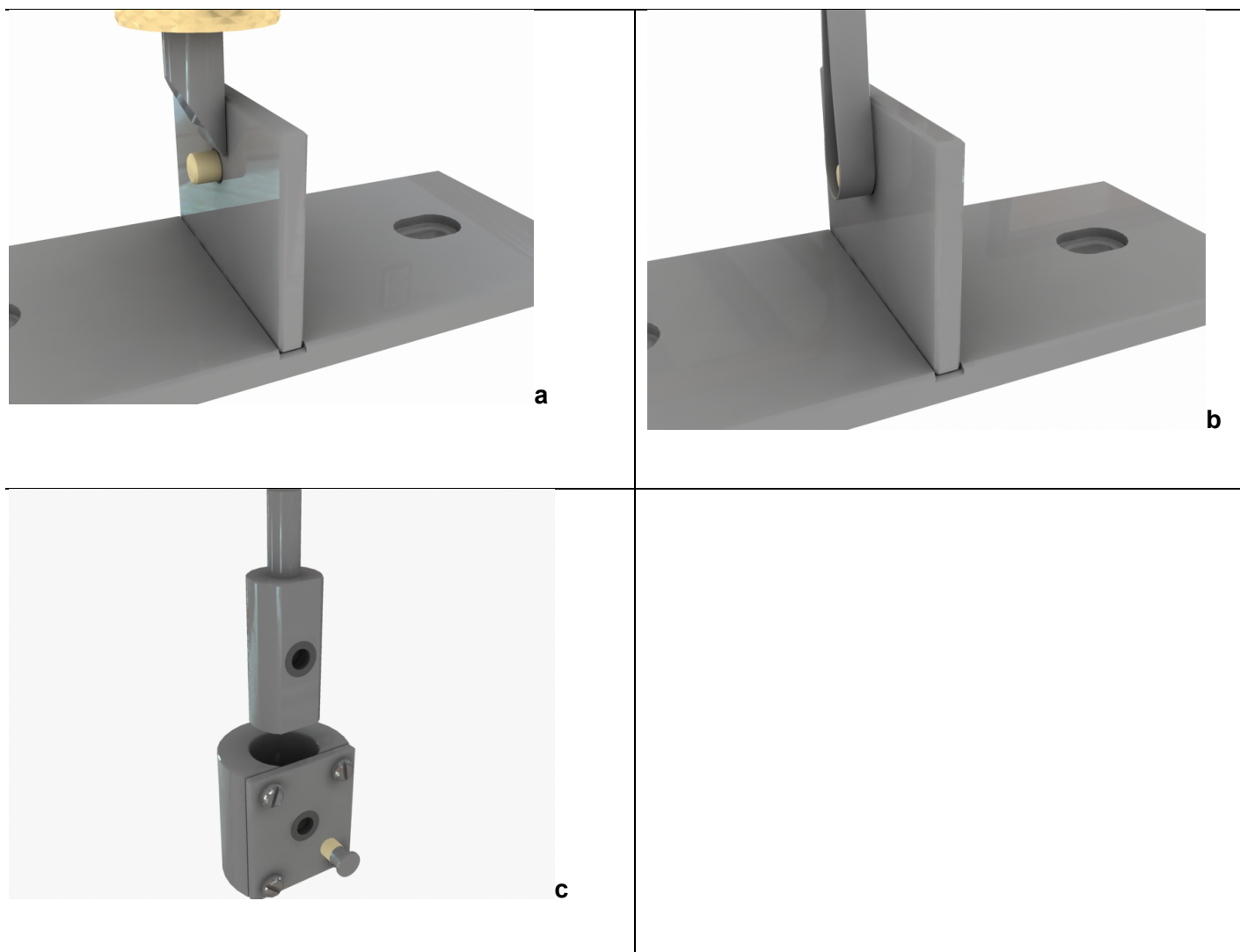
Figs. 1a-c The jig designs used for testing adhesion of veneering ceramic to metal **a)** chisel (CH) (ISO 11405) (blade thickness: 1mm), **b)** steel strip (SS) (width: 5 mm, length: 1 cm), **c)** piston (PI) allowing the placement of the metallic part of the specimen in the external part and the veneering ceramic in the internal part.

Fig. 2 Means and standard deviations of bond strength of veneering ceramic to metal using different types of jigs. **a)** Chisel (CH), **b)** Steel strip (SS), **c)** Piston (PI).

Figs. 3a-i **a)** Metal surface after debonding using CH where the presence of opaque ceramic at the margin could be observed (m: metal; c: ceramic), **b-c)** SEM images (x75 and x2200) from this group showing that the failure occurred in the ceramic located in the region where the chisel was applied, **d)** Metal surface after debonding using SS jig. Note that larger quantity of ceramic remained on the metal in the form of a half-moon fracture compared to other groups, **e-f)** SEM images (x33 and x1300) showing the cohesive fracture in the ceramic initiating from the margin, **g)** Metal surface after debonding using PI jig showing less ceramic remnants, **h-i)** SEM images (x75 and x1700) showing extensive areas of metal indicating adhesive interfacial failure. The principle load direction (F) is indicated with an arrow in each image.

Figs. 4a-c FEA analysis of test simulation from lateral, front view of metal-ceramic assembly, transversal section of metallic base and ceramic surface when shear force was applied and tests were performed using different types of jigs. Note the highest stress concentration with the CH followed by SS and PI. For interpretation of the PI group, it should be noted that mathematical model was performed with perfect fit. The symmetry of the loading pattern in the figures are unexpected as there should be some distortion as the loading occurs which could be due to the hole in the piston that does not have a precise fit to the specimen. The principle load direction (F) is indicated with an arrow in each image.

Figures:



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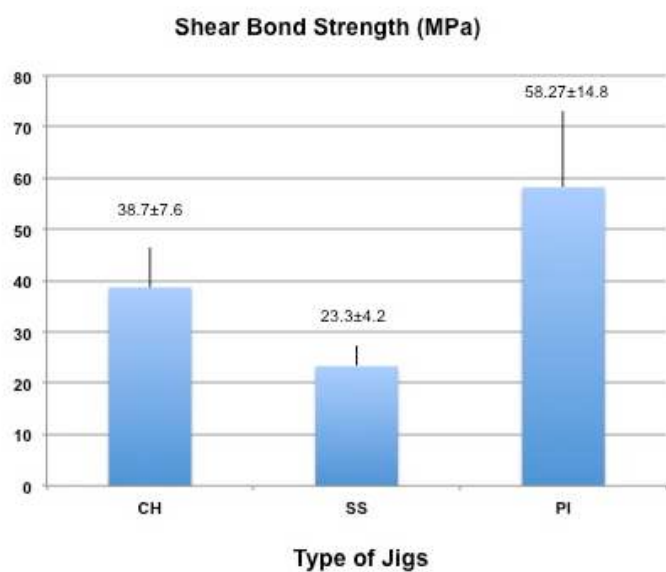
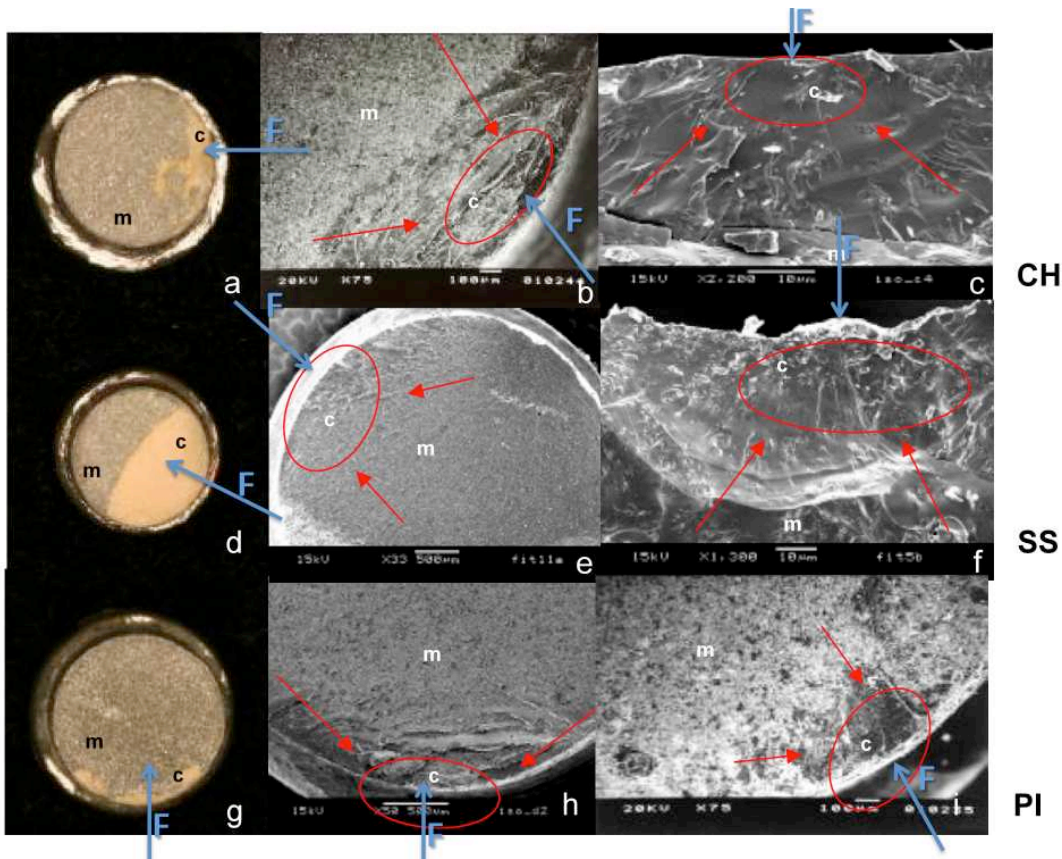
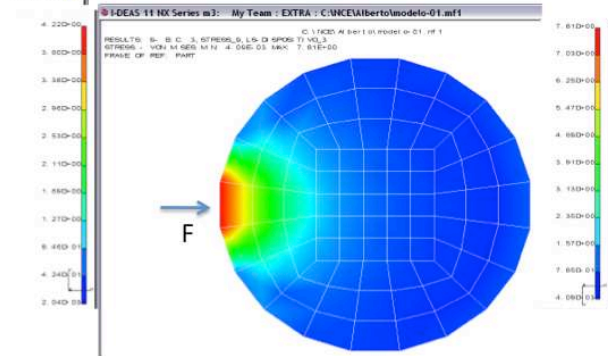
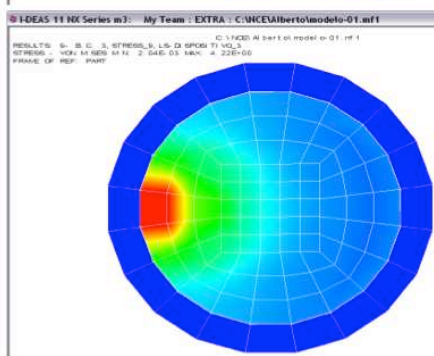
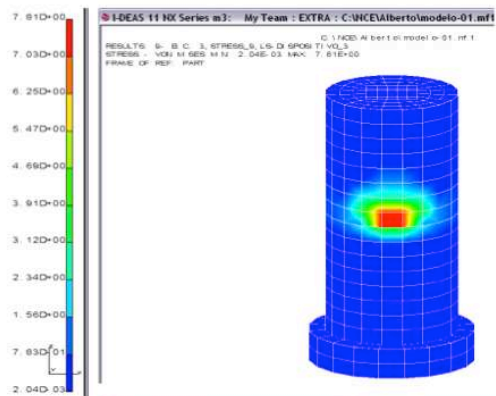
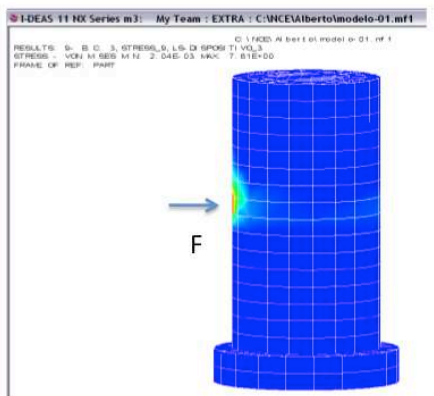


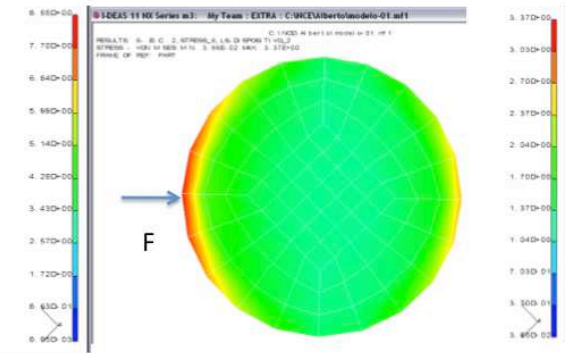
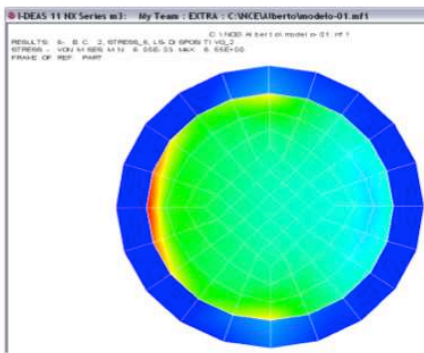
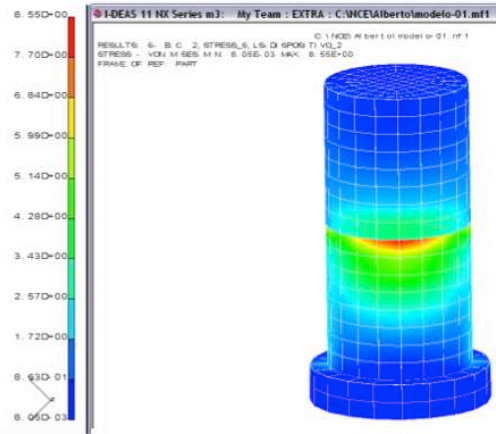
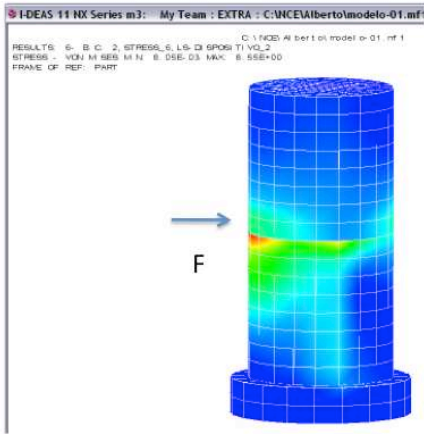
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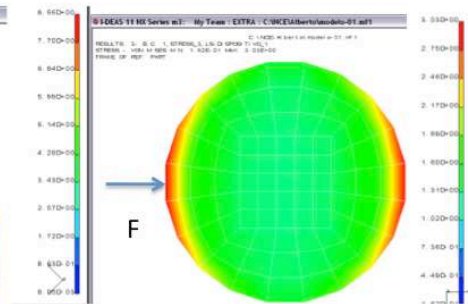
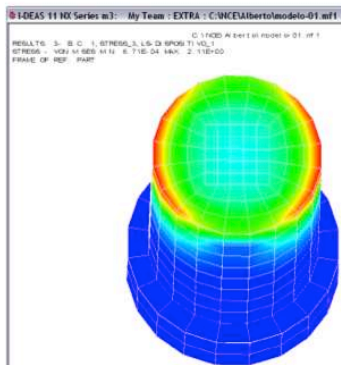
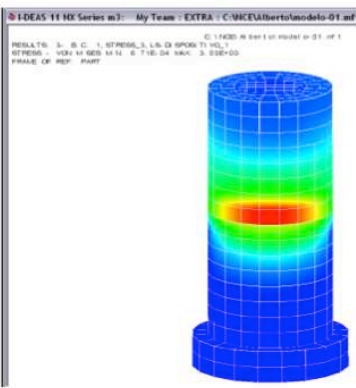
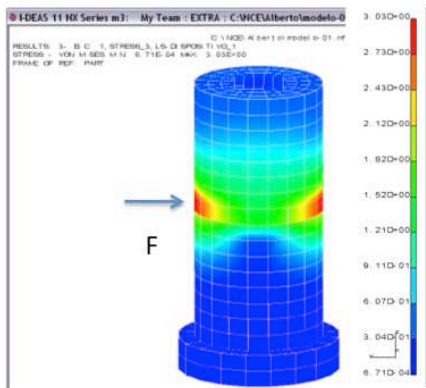
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CH



SS



PI

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